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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of: Alvin S. Williams

Confirmation No: 2210

Application No.: 10/792,375

Group Art Unit: 1743

Filed: March 2, 2004

Examiner: Monique T. Cole

For: ODORANT COMPOUNDS

Attorney Docket No.: 81455-5870

RULE 132 DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Pierre-Alain Blanc, hereby submit the following comments as a person of ordinary skill in the art of the subject matter of this application:

1. I have a doctorate in Chemistry and over 26 years experience as perfumer. I am presently working at Firmenich, SA, the assignee of the present application. Over the past twenty-two years, I have been actively involved in research regarding the use of emulsions in perfuming compositions and the evaluation of such emulsions. I currently hold the position of chairman of an evaluation panel of expert and my specific duties include the assessment of the interest and commercial potential of new fragrance materials.

2. I am familiar with the present invention, its claims and the office actions that include rejections of such claims.

3. The present invention relates to compounds that when added to a perfuming composition or perfume, the compound provides a musky-green odor character note. In particular the green character imparted by these compounds is a fresh note having a Galbanum (see the attached description of the Galbanum odor) and green-pear's peel connotation. Furthermore, to the best of my knowledge the invention compound is unique in the sense that no

other known compound combine a musky-ambrette note with a green note.

For the sake of clarity it can be useful to mention that by green character it is intended in the art a note having a typical foliage/herbaceous and acidic character.

4. I understand that the claims were rejected as being unpatentable over U.S. Patents No. 5,166,412 to Giersch et al, which disclose the compound 4-(3,3-dimethyl-1-cyclohexyl)-2,2-dimethyl-3-oxapentyl propionate.

5. The office action assumes that since the present compounds differ from that of Giersch only by the presence of a double bond there is a presumed expectation of similar properties because the compounds are homologues. This presumption is incorrect because these compounds have significantly different properties and utilities. Although both compounds are useful as perfuming ingredients, the present compounds have distinctly different odor properties and organoleptic utilities.

6. The prior art compound 4-(3,3-dimethyl-1-cyclohexyl)-2,2-dimethyl-3- possess musky odor as well as a floral undertone and a fruity character of the pear type, i.e. has a combination of the ambrette and fruity-pear character. The musky character becomes even more important in the case of the optically active compounds. For the sake of clarity it can be useful to mention that by fruity character it is intended in the art a note having a typical sweet character.

7. Therefore, the character of the odor properties of the present compounds differs from the ones of the prior art by having a green-Galbanum character. This green note is a foliage/acidic note and is totally absent from the odor of the prior art compound which possesses a fruity/sweet note. The odor character of the compounds of the present invention is defined in the specification in paragraphs [0012] to [0014] and is unique in the sense that combines a musky and green odor in a single compound. In addition, the odor is very diffusive and this is rare for a compounds that possess a musky note. These differences are all the more surprising and unexpected in view of the compounds disclosed in Giersch. They certainly are not obvious or extrapolatable from the prior art compound described in Giersch. Indeed, there is nothing in Giersch that leads a skilled artisan to foresee the presently claimed odor character simply based on the structural similarity of the prior art compound.

8. There is no doubt in my mind whatsoever, that the compound discovered by us, e.g. 4-(3,3-dimethyl-1-cyclohexyl)-2,2-dimethyl-3-oxapentyl propenoate, has an odor which is different from that of the known methyl 4-(3,3-dimethyl-1-cyclohexyl)-2,2-dimethyl-3-oxapentyl propionate, in spite of the fact that they have very close structures. These two compounds, as such, are well suited for different applications, indeed the present compound (I) is particularly well fitted to be incorporated into masculine, aromatic or citrus type of preparation while the prior art compound is more fitted to be incorporated into feminine, oriental type of preparation.

9. In this art, a skilled artisan cannot rely on structural closeness to predict the organoleptic characteristics of a specific compound, or the usefulness of the odor properties of the compound. Therefore, although the prior art and present compounds are related as structural homologues differing in the position of the double bond, the compounds are actually of different nature, with different properties and organoleptic utilities, and any presumption of property similarities between the compounds should be overcome.

I further declare that all statements made in this declaration of my own knowledge are true and that all statements made on information and belief are believed to be true ; and further these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 13th day of June, 2006



Pierre-Alain Blanc



Perfume and Flavor Materials of Natural Origin

By

STEFFEN ARCTANDER

1960

ELIZABETH, N.J. (U.S.A.)

FIRMENICH & Co
GENÈVE

E 234

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Galanga rhizomes have since long been used
as a non-pungent additive in the production of
Chinese "preserved stem ginger" which is exported
in the very decorative "bojans" (artistically hand-
painted and ornamented clay or china jars).

See also Ginger Absolute, Ginger Oil, Ginger
Oleoresin.

Galbanum.

Galbanum is a natural oleo-gum-resin (see defini-
tions in Part One of this work). Speaking strictly
in accordance with our definitions, galbanum
should be listed as a natural oleo-resin since the
perfumery grade of galbanum contains very little
gum.

Galbanum is one of the typical examples of a
botanical which has "changed" appearance, com-
position and odor during the past five decades.
This is apparent when we look into literature
from 1939, 1936, 1926 or even further back: the
description of galbanum in these works is out-
dated. A few, more modern authors suggest that
more rapid transportation to the user is the main
reason for the significant change, but it seems
inconceivable that this should be the only reason.
The author of this book has seen various African
and Middle-eastern "gums" arrive at the original
shipping stations after harvesting, and it appears
that the time which elapses between harvesting
and the arrival of the merchandise on board ship
in the nearest export harbor, has not changed
significantly. Only the transportation from the
port of origin (departure) to the destination (user)
has been slightly shortened.

Galbanum is collected from not one, but several
different species of *Ferula* (big umbellifer plants).
These grow wild in Iran and in the countries
toward Asia Minor, e.g. Lebanon. There are
still—as the literature correctly says—two types:
a hard and a soft galbanum offered commercially.
For perfumery, only the soft variety has interest.
The hard galbanum is used in pharmacy and for
various industrial purposes.

Soft Galbanum is a dark amber-colored to
yellowish-brown or grayish-green, olive-brown,
very viscous liquid whose consistency is like fresh
honey. It is usually contaminated with wood
splinters, sand, gravel, plant fibres, insects, etc.,
and it will separate a "foot" of a grayish mass on
the bottom of its container. This precipitate

contains water, sand, some dissolved gum, etc.,
and it has no perfumery value. It is often possible
to eliminate it when emptying a container, since
the precipitate is not pourable, while the useful
galbanum is just pourable at room temperature
(over 20°C.).

The odor of crude galbanum (soft type) is
powerful, green-woody, almost balsamic-resinous,
reminiscent of oleoresins from conifers. The leafy-
earthy, green note is often referred to in terms,
such as: "like green peppers" or "tossed green
salad", etc. There is a distinct resemblance to the
odor of hyacinth leaves. For a further odor
description, see Galbanum Resinoid.

The "hard" galbanum will be discussed here
only with respect to its physical description: it is
a dry, solid, gravel-like material with a faint odor
and variable color; it consists of yellow, orange,
red tears or drops, and it is somewhat less hard and
brittle than olibanum which it resembles to a
certain degree. This type of galbanum is known as
Persian Galbanum.

Soft Galbanum or Levant Galbanum is collected
in very large quantities, and tens of tons are used
by the perfume industry, in contradiction to
various statements that "galbanum is used to a
limited extent in certain perfume types". It is true
that its application is limited to a comparatively
small number of perfume types, but some of these
types are very common in use. The Soft Galbanum
also serves as a starting material for the distilla-
tion of Galbanum Oil (see monograph) which is
abundantly present in this type of galbanum.

Being an exudation of a physiological (naturally
formed) product, Galbanum does not require much
work beyond its actual collection. It is conceivable
that the perfumery type of galbanum is mainly
derived from those species of *Ferula* in which the
exudation appears at the umbel itself. This exuda-
tion contains more essential oil, and is softer than
the exudations from stalks, etc. from other species
of *Ferula*.

See monographs on Galbanum Oil and Galb-
anum Resinoid.

Galbanum Oil.

Galbanum oil is steam distilled or steam-and-
water distilled from Galbanum (see previous mono-
graph). The "soft" or "Levant" type of galbanum
is preferred since it contains far more essential oil.
The yield of Galbanum Oil by steam/water distilla-

tion is usually in excess of 15%, often around 22%, calculated upon the weight of the crude botanical material. The author has obtained yields of 26% of galbanum oil from good qualities of galbanum. Distillation is undertaken far away from the origin of Galbanum; usually the oil is distilled in France, Germany, England or in the U.S.A., and it is quite customary for perfume houses to distil their own galbanum oil. As a rule, certain fractions of the distillate are eliminated prior to bulking of the essential oil. Sulfide odors in the light fractions (heads) and strong painty-terpene notes in the tail fractions (and in the medium-light fractions), particularly those rich in alpha- and beta-pinene, are usually left out. A partially dewatered (monoterpene-free) oil is known as Galbanol. There is no actual standard as to the composition of galbanum oil or galbanol, and the selection of fractions is primarily a matter of esthetics.

Galbanum Oil is a colorless to pale yellow or pale olive-yellow, mobile liquid which possesses an intensely green, fresh-leafy odor with a dry-woody undertone of balsamic, barklike character. A very striking description often heard is "like green peppers or tossed green salad". The oil has a pine-like topnote which is less pronounced in the odor of the resinoid. The latter, in turn, has a more woody-balsamic, conifer-resinous character. The pine-like topnote can be removed by fractional distillation of the oil. The woody-balsamic, soft-green and tenacious undertone can be isolated in the high-boiling fractions. Galbanol is a trade name for the latter type of galbanum oil fractions.

Galbanum oil finds extensive use, although always in very modest concentrations, in compositions of chypre, fougère, pine, forest, moss, etc., and it will introduce interesting notes in many florals where its leafy character is necessary in the completion of a true naturalness: hyacinth, violet, narcissus, lavender, gardenia, etc. It blends well with cinnamic alcohol, coumarin, cuminaldehyde, dimethyl benzyl carbinol, geraniol, geranium oil, cyclamal, isoeugenol, linalool, oak-moss products, pine needle oils, fir needle absolute, methyl phenyl carbonyl propionate, styrax resinoid or styrax oil, etc., and it can actually find use in countless perfume types and bases.

The annual production of galbanum oil fluctuates to quite a degree, but it is steadily increasing, possibly exceeding 10 metric tons. Adulteration is not uncommon, usually by means of simple

dilution with pinene, foreruns from "galbanol" (see above), camphene, etc. These additions will easily be detected by the experienced perfumer during an olfactory examination of the oil.

Galbanum Resinoid.

Galbanum resinoid is prepared from the crude galbanum (see monograph). Years ago, when galbanum was a hard, grainy mass or lumps, the resinoid was prepared in the conventional way, i.e. by extraction with a hydrocarbon solvent and subsequent removal of the solvent after filtration. Because of the significant water content in the lower grades of galbanum, it was necessary to use solvents which are not miscible with water. Acetone and ethyl alcohol could not be used.

During the 1950's, it became more and more "conventional" to prepare resinoids in such a way, that they would be pourable, regardless of the starting material from which they were derived. This was done by the simple addition of a solvent to the evaporation residue (which is the pure, "100%" resinoid). The customer had the advantage of getting a more handy material; weighing and mixing was easier, etc. This dilution idea was further developed into more practical extraction methods: an odorless, colorless, high-boiling solvent (or "plasticizer") was added during the evaporation, and it was left in the finished product which was just pourable at room temperature. Various botanical raw materials called for different amounts of solvent to be added according to the viscosity of the "100%" (i.e. solvent-free) resinoid. The content of essential oil in the resinoid determines the viscosity of the solvent-free resinoid. In exceptional cases, where the content of essential oil is so high that the botanical material is almost liquid (a so-called "balsam"), the above method can be revised to a simple addition of a non-distillable solvent to the crude botanical material. This mixture can be filtered or strained and the "resinoid" is ready for use.

Crude Galbanum can be mixed with a certain amount of diethyl phthalate, isopropylmyristate, isopropylpalmitate, diethyl sebacate or similar solvent under gentle heating. Water and dirt will rise to the surface, respectively fall to the bottom of the mixture. After filtration or straining, the dregs, etc. are usually extracted with a volatile solvent. The extract is filtered and evaporated. The residue is added to the above prepared solu-

tion of galbanum benzoate was used. It contributes to the plasticized resinoid or months during of precipitate will may also separate soluble in all type glycol or similar product should be on the amount of Resinoid 67%, in

However, the "sold under various be used exclusively obviously carries can no longer material to evaluate essential oil. The and it is so in many by other material depressant, and it "galbanum-solvent resinoid (solvent-willing to mention and solvent in his

Solvent-free gal dark amber or be pourable at room ly rich-green, w undertone, and it foliage-like note essential oil (se conifer-balsamic resinoid and in resinoid is usually. Extracts of galban as a solvent, are a are almost clearl more or less "te different odor typ balsamic, rich, w

Galbanum Resin fixative with an lavender, fougère fragrances, wood certain floral type classic. Galbanu advantage over materials in the immediately: it is

from "galbanol" these additions will be experienced by the perfumer of the oil.

from the crude years ago, when mass or lumps, the conventional way, carbon solvent and not after filtration. or content in the is necessary to use with water. Acetone used.

more and more resins in such a way, regardless of which they were a simple addition residue (which is the customer had a handy material; etc. This dilution is more practical, colorless, high-purity was added during the finished at room temperatures materials called for be added according (i.e. solvent-free) oil in the resinoid solvent-free resinoid re the content of botanical material (sam"), the above simple addition of a crude botanical filtered or strained use.

ed with a certain propylmyristate, acetate or similar ester and dirt will fall to the bottom or straining, the oil with a volatile and evaporated. the prepared solu-

tion of galbanum in an odorless solvent. Benzyl benzoate was used some years ago, but it contributes to the odor in an unwanted way. The plasticized resinoid is left aside for several weeks or months during which period a certain amount of precipitate will settle. Small amounts of water may also separate. The finished product is now soluble in all types of perfume materials, but not clearly soluble in diluted alcohol, propylene glycol or similar hydrophilic solvent types. The product should be labelled with clear information on the amount of solvent added, e.g.: "Galbanum Resinoid 67%, in D.E.P." or the like.

However, the "prepared" resinoids are usually sold under various trade names or they may often be used exclusively by the producer. The method obviously carries a certain hazard: The customer can no longer rely upon the viscosity of the material to evaluate the approximate content of essential oil. The oil can be partially removed—and it is so in many cases—or it can be replaced by other materials. The solvent acts as an odor-depressant, and it is difficult to evaluate the above "galbanum-solution" in comparison to a true resinoid (solvent-free), even if the supplier is willing to mention the exact percentage of resinoid and solvent in his product.

Solvent-free galbanum resinoid is a semi-liquid, dark amber or brownish-golden material, almost pourable at room temperature. The odor is intensely rich-green, woody-balsamic, yet with a dry undertone, and it has the typical "green peppers" foliage-like note which is so pronounced in the essential oil (see monograph). The resinous, conifer-balsamic notes are very pronounced in the resinoid and in the prepared solutions. The resinoid is usually not clearly soluble in alcohol. Extracts of galbanum, prepared with ethyl alcohol as a solvent, are available. These "resin absolutes" are almost clearly soluble in alcohol. They are more or less "terpeneless" products of entirely different odor type: less green-sharp, more soft-balsamic, rich, woody and very tenacious in odor.

Galbanum Resinoid is an extremely interesting fixative with an odor of its own. It is useful in lavender, fougère, Oriental bases, chypres, pine fragrances, woody bases, moss odors, and in certain floral types. Its use in hyacinth is almost classic. Galbanum resinoid has the definite advantage over many synthetic "green-odor" materials in the fact that it mellows in almost immediately: it is possible to evaluate the result

and the effect during the creation of the fragrance. Synthetic materials in this odor group often "grow" or "fade" in the perfume with a perceptible change in the odor of the perfume within a few weeks.

The annual world production of Galbanum Resinoid is adjusted to the demand which has increased enormously during the past decade (1950's). So far, there has been no shortage of this material.

Gardenia.

The gardenia shrub, *Gardenia Grandiflora* (and other gardenias), is quite common as an ornamental plant in subtropical and warm-temperate zones of the world. The flowers of this plant have been known and admired for their outstanding fragrance for thousands of years. However, these flowers are rarely submitted to extraction for the isolation of essential oil, concrete or absolute. One reason is the very small yield (about 1 kilo of absolute from 5000 kilos of flowers). Another reason is the limited use of the gardenia type of fragrance in perfumery. Finally, this type of fragrance has been comparatively easy to copy, although a good artificial gardenia base is more rare than for example a good artificial muguet base (lily-of-the-valley). Incidentally, a close resemblance to the natural product is not synonymous with unexcelled performance in a perfume.

A concrete of Gardenia flowers was produced many years ago in the Indian Ocean island of La Réunion when the French extraction expert, Charles Garnier established himself with his world-famous equipment on that island. Production in La Réunion has been abandoned long ago. Various sorts of *Gardenia Absolutes* are occasionally offered on the market today. Some of these may actually derive from Grasse factories, but quite recently, Chinese and Formosan producers have offered *Gardenia Concrète* in Europe and India.

The Chinese gardenia concrete is presumably derived from the flowers of *Gardenia Florida* which is a native of southeastern China. *Gardenia Grandiflora* is grown in China for its fruits which yield Wong-Shi, a yellow colorant. Other varieties grow in Japan, in the Philippines, Indonesia, India, the West Indies, etc., but they are rarely utilized for perfume extraction.

It serves no purpose to describe here outdated

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